

Frequency dependence of aging, rejuvenation and memory in a disordered ferroelectric

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Abstract. We characterize in details the aging properties of the ferroelectric phase of $\text{KTa}_{1-x}\text{Nb}_x\text{O}_3$ (KTN), where both rejuvenation and (partial) memory are observed. In particular, we carefully examine the frequency dependence of several quantities that characterize aging, rejuvenation and memory. We find a marked subaging behaviour, with an a.c. dielectric susceptibility scaling as $\omega\sqrt{t_w}$, where t_w is the waiting time. We suggest an interpretation in terms of pinned domain walls, much along the lines proposed for aging in a disordered ferromagnet, where *both* domain wall reconformations and overall (cumulative) domain growth are needed to rationalize the experimental findings.

PACS. 05.40.-a Fluctuation phenomena, random processes, noise, and Brownian motion – 77.22.Gm Dielectric loss and relaxation – 78.30.Ly Disordered solids

1 Introduction

Aging is a widespread phenomenon which manifests itself through the dependence of the properties of a material on the history of the studied sample [1]. It has been observed and widely documented on the magnetic susceptibility of spin-glasses (SG) [2–5] and disordered ferromagnets [6], on the elastic compliance of polymers [7], on the dielectric constant of disordered dielectrics ('dipolar glasses') [8,9] and of some liquids [11], and recently on the rheological properties and dynamical structure factor of 'pasty' colloids [12,13]. Aging reflects the fact that the time needed for the system to equilibrate becomes larger than the experimental time scales. Although several theoretical models for aging have been investigated, the correct physical picture for aging is still rather controversial, in particular in spin-glasses. The simplest model for aging is that of domain growth: below some critical temperature, the system progressively orders by growing domains of the competing low temperature phases (for example up and down in the case of an Ising ferromagnet). In this case, the aging of the susceptibility can be understood from the dynamics of pinned domain walls: as time elapses, the density of domain walls decreases, and the remaining domain walls are better and better pinned.

Stronger constraints on microscopic theories appear when one wants to interpret the so-called 'rejuvenation

and memory' effect [14]. Suppose that one cools a glassy system at a constant cooling rate \mathcal{R} down to a certain temperature T_{pl} , which is then held constant. At this temperature, aging manifests itself by the decrease of some susceptibility (both of its real and imaginary part). If cooling is then resumed after a 'plateau time' t_w , the susceptibility is seen to increase again, approaching the value it would have had if t_w was zero: the system 'rejuvenates' as it seems to forget its stay at T_{pl} . If cooling is carried further, down to low temperatures and then followed by a steady heating, a 'dip' in the susceptibility is observed when passing back through the temperature T_{pl} . This shows that the system has actually kept some 'memory' of its evolution at T_{pl} . The coexistence of these two effects is quite remarkable, and has led to several theoretical scenarios [15–18]. These two effects have been seen in a variety of materials: various spin-glasses, PMMA (plexiglass) [19] or KTN (a disordered dielectric, see below) [20]. However, some materials exhibit aging but no rejuvenation [21], or rejuvenation but no (or partial) memory [6].

The aim of this paper is to characterize in more details the aging properties of the ferroelectric phase of KTN, where both rejuvenation and (partial) memory are observed [20]. In particular, we have carefully examined the frequency dependence of several quantities which characterize aging, rejuvenation and memory. We suggest an interpretation in terms of pinned domain walls, much along the lines proposed in [6] for a disordered ferromagnet, where both domain wall *reconformations* and overall

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domain growth are needed to rationalize the experimental findings.

Aging has been already observed in ferroelectrics. For instance, aging in the ferroelectric lock-in phase of Rb_2ZnCl_4 [22] was attributed to wall trapping and to an increase of the total wall area; moreover, it was noticed that the effects are dispersive. Another example is provided by solid solutions of lead magnesium niobate and lead titanate doped with a small amount of manganese oxide MnO [23]. This material, which has a perovskite structure, exhibits aging and rejuvenation (even if the word is not used); here too the effects are dispersive. However, in these examples the frequency dependence was not quantitatively studied, no memory was seen and therefore the relationship between aging and memory was not discussed.

2 Experiments

The pure potassium tantalate crystal KTaO_3 belongs to the cubic perovskite family. Quantum fluctuations prevents a ferroelectric order to establish fully, even at $T = 0$ K. If a fraction x of tantalum ions is randomly substituted by isoelectronic niobium ions to give crystals of $\text{K Ta}_{1-x}\text{Nb}_x\text{O}_3$ (KTN), the trend towards ferroelectricity of the perovskite lattice is enhanced. It has been assumed that this tendency was due to random fields generated by the niobium ions [25]. Indeed, if the niobium concentration x is larger than $x_c = 0.008$, the cooperative ordering gives way to a ferroelectric phase at temperatures below a finite transition temperature T_c . For the sample used in the present study we observe a broad transition at $T_c = 31$ K, defined by the maxima of the real part ϵ' and the imaginary part ϵ'' of the dielectric constant, independently of the measuring angular frequency ω . When $x > x_c$, there are actually several transitions: when the temperature is lowered, KTN is successively cubic, tetragonal, orthorhombic and finally, rhombohedral [26]. For the concentration x of our sample, the three transitions merge into a single one. From the phase diagram obtained in [26], we infer that the niobium concentration is around $x = 0.022$. The experiments reported below were all performed at temperatures $T < 25$ K, where the material has a local rhombohedral symmetry but is macroscopically cubic, and is in an ordered (ferroelectric) phase which contains some amount of disorder (the randomly substituted niobium ions).

Using a Hewlett-Packard 4192A impedance analyser, we have measured the electric capacitance and the dielectric loss at seven frequencies, ranging from $f = 1$ kHz to $f = 1$ MHz. These can easily be transformed into the real part ϵ' and the imaginary part ϵ'' of the complex dielectric constant: $C = 1$ pF corresponds to $\epsilon_r = 16$. The electric field is of the order of 1 kV/m, such that non linearities can be neglected, at least for $T < 20$ K. Non linear effects do become strong around the transition temperature T_c . Note that our fields are much weaker than those used in [24] (ranging from 2 to 200 kV/m) for a study of the non linear dielectric constant of a relaxor ferroelectric, where 2 kV/m is already considered as a very weak field.

We measure the complex capacitance $C(\omega, t, T)$ as a function of time t while the sample temperature T is a controlled function of time. Several procedures were used after an annealing near 55 K and an initial rapid cooling across the transition temperature T_c down to $T_{\text{max}} = 22$ K. The simplest procedure was composed of a cooling at a constant rate $dT/dt = -\mathcal{R}$ from T_{max} to $T_{\text{min}} = 4.8$ K, immediately followed by a steady heating at the opposite rate $dT/dt = +\mathcal{R}$ from T_{min} to T_{max} : this was used as the *reference curves*. Another procedure is to cool at the same rate $-\mathcal{R}$ between T_{max} and an intermediate temperature T_{pl} . There, cooling is interrupted by an isothermal evolution or plateau at this temperature, which lasts typically $t_w = 10\,000$ seconds. Cooling then resumes down to T_{min} and is again immediately followed by a steady heating at the opposite rate from T_{min} to T_{max} . A typical cooling rate is $\mathcal{R} = 5.2$ mK/s.

3 Frequency analysis of isothermal aging

The isothermal variation of the complex capacitance $C(\omega, t)$ in KTN is a slow decay in time (which also depends on the cooling rate \mathcal{R} [27]). This decay can be fitted by a power law $(t_0 + t)^{-\eta}$ with a small exponent η (typically $\simeq 0.05$) and t_0 in the range of 10^2 s. For a given plateau temperature T_{pl} and a given plateau duration t_w , the total decay of the complex capacitance, $\delta C(\omega, t_w, T_{\text{pl}}) \equiv C(\omega, t = 0^+, T_{\text{pl}}) - C(\omega, t = t_w, T_{\text{pl}}) \geq 0$ is found to obey a negative power law of the frequency f and therefore of $\omega = 2\pi f$. The real part and the imaginary part of this variation can be fitted by (see Fig. 1):

$$\delta C'(\omega, t_w, T_{\text{pl}}) = N' \omega^{-\nu} \quad \delta C''(\omega, t_w, T_{\text{pl}}) = N'' \omega^{-\nu}, \quad (1)$$

with the same exponent ν . This is actually a general consequence of the Kramers-Krönig relations, which hold for a linear, stationary and causal response theory. Although aging means that the response is non stationary, these Kramers-Krönig relations are valid in the quasi stationary limit where $\omega^{-1} \ll t_w$, which obviously holds in our experiments (where $\omega t_w \gg 10^3$). The Kramers-Krönig equations actually also provide a precise relation between the amplitudes N' and N'' , which only depends on the value of ν :

$$\frac{N''}{N'} = \tan\left(\frac{\nu\pi}{2}\right). \quad (2)$$

This relation provides a second determination of ν , by checking the constancy of the ratio of the imaginary and real parts of δC , and using equation (2) to extract ν . As shown in Figure 1, obtained for $T_{\text{pl}} = 12.1$ K and $t_w = 20\,000$ s, these two procedures allow one to obtain rather close estimates for ν . The regression in log-scale based on equation (1) gives $\nu = 0.127 \pm 0.005$. The exponent ν is found to depend weakly both on temperature and time. More precisely, it varies linearly with T_{pl} but only

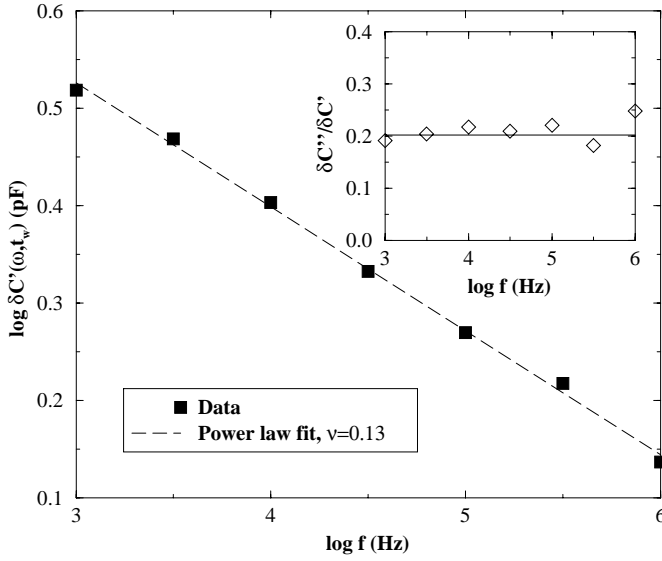


Fig. 1. Behaviour of the total decay of the real-part of the capacitance, $\delta C'(\omega, t_w, T_{\text{pl}}) \equiv C'(\omega, t = 0^+, T_{\text{pl}}) - C'(\omega, t = t_w, T_{\text{pl}})$, as a function of the frequency $f = \omega/2\pi$, for $t_w = 20\,000$ s and $T_{\text{pl}} = 12.1$ K. The magnitude of this decay is found to behave as a power-law of the frequency: $\delta C' \propto \omega^{-\nu}$, with $\nu = 0.127 \pm 0.005$ over three decades in frequency (1 kHz – 1 MHz). We show in the inset the ratio $\delta C''/\delta C'$ as a function of frequency. For a power-law dependence, this ratio should be constant, equal to $\tan(\nu\pi/2) = 0.202$, shown as the full line.

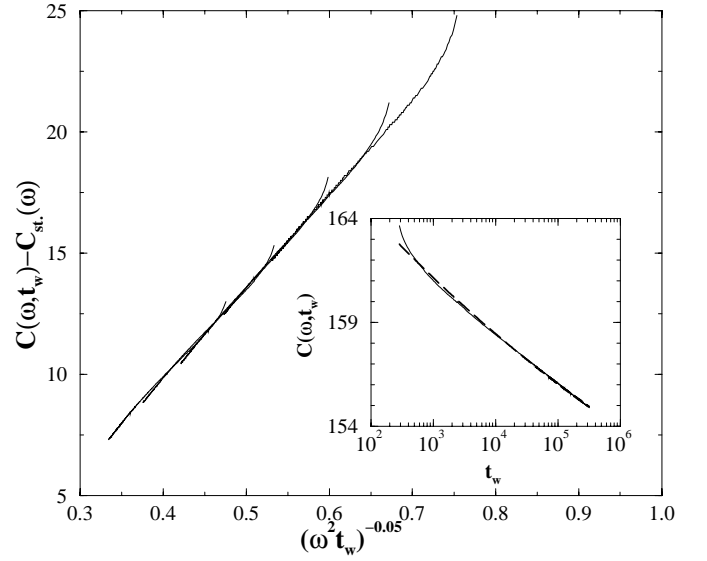


Fig. 2. Rescaling of the capacitance for various frequencies ω (from 1 kHz to 1 MHz) and plateau times t_w . The temperature here is $T_{\text{pl}} = 9.8$ K. We have chosen the stationary part $C_{\text{st}}(\omega)$ (corresponding to a vertical shift) to obtain the best rescaling, as a function of $(\omega^{1/\mu} t_w)^{-\eta}$, with $\mu = 1/2$ and $\eta = 0.05$, corresponding to $\nu = 0.10$. The rescaling is not perfect for the early time part where the time scale t_0 introduced in the text cannot be neglected. Inset: Relaxation of $C(\omega, t_w)$ at 3.16 kHz, and fit of the decay with a pure power law of exponent $\eta = 0.05$ (dashed line).

logarithmically with t_w . The magnitudes N' and N'' are increasing functions of T_{pl} and t_w .

In spin glasses, the dependencies of the aging part of the susceptibility on the waiting time t_w and on the frequency ω are not independent, but rather follow a simple ωt_w scaling, which means that the typical relaxation time after waiting t_w is t_w itself. More generally, one may expect scaling with ωt_w^μ , where μ is an exponent, generally found to be smaller than 1: this corresponds to ‘sub-aging’ [1, 16]. It is interesting to look for a similar scaling in our data. As shown in Figure 2, the aging part of the capacitance can indeed be approximately rescaled with $\mu \simeq 0.5$, leading to:

$$C(\omega, t_w, T_{\text{pl}}) \simeq C_{\text{st}}(\omega, T_{\text{pl}}) + \mathcal{C}(T_{\text{pl}}) \times (\omega t_w^\mu)^{-\nu} \quad t_w \gg t_0, \quad (3)$$

where C_{st} is the stationary part of the response. This equation describes simultaneously both the above discussed frequency dependence and the time relaxation of the aging part, with the exponent relation $\eta = \nu\mu$.

4 Frequency analysis of rejuvenation and memory

A useful way to parametrize the ‘strength’ of rejuvenation is to compare the slope $\mathcal{S} = \partial C'/\partial T$ of the capacitance with temperature just after the plateau at T_{pl} with the slope \mathcal{S}_r of the reference curve, which is positive for all

frequencies. The stronger the difference $\delta\mathcal{S} = \mathcal{S}_r - \mathcal{S}$, the stronger the ‘rejuvenation’ effect. We find again that $\delta\mathcal{S}$ is a negative power-law of the frequency: small frequencies are more efficiently ‘rejuvenated’. The value of the exponent is found to be similar to that of ν . For example, for $T_{\text{pl}} = 14.3$ K and $t_w = 10\,000$ s, we find an exponent equal to 0.20. Note that for small enough frequencies, the capacitance increases when the temperature is decreased ($\mathcal{S} < 0$), while the capacitance decreases ($\mathcal{S} > 0$) at high frequencies, as is the case of the reference curve.

Figure 3 shows, as a function of temperature, the real part of the capacitance $C'(\omega, T)$ for two thermal histories. The first curve is the reference curve corresponding to a regular cooling from T_{max} to T_{min} , followed by a regular heating from T_{min} to T_{max} . The value of $C'(\omega, T_{\text{min}})$ along this path is indicated by point ‘C’ in Figure 3, and the value of $C'(\omega, T_{\text{pl}})$ at $T = T_{\text{pl}}$ are point ‘A’ on the cooling curve and point ‘E’ on the heating part.

The second curve corresponds to a regular cooling from T_{max} to T_{pl} (point ‘A’ again), followed by isothermal aging at $T_{\text{pl}} = 12.1$ K for a certain time $t_w = 20\,000$ s, reaching point ‘B’. The isothermal decay reported above is therefore $\delta C'(\omega, t_w, T_{\text{pl}}) = |A - B|$. Then regular cooling resumes from T_{pl} to T_{min} , where it reaches point ‘D’ which is *below* point ‘C’. The distance $|C - D|$ reflects some cumulative aging due to the plateau at T_{pl} . The last part of the curve is again obtained by a regular heating of the system. The value of $C'(\omega, T_{\text{pl}})$ when passing T_{pl} is point ‘F’.

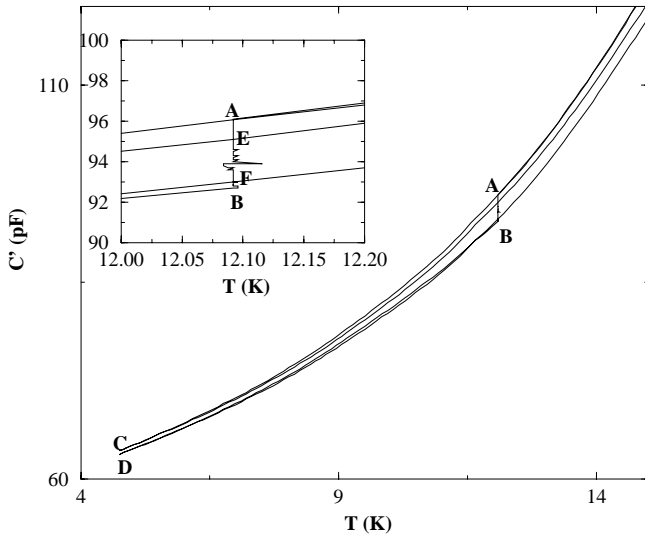


Fig. 3. Capacitance $C'(\omega, T)$ at 1 kHz, for two thermal histories, one without a plateau at T_{pl} , the other with a long plateau at T_{pl} . The important points A, B, \dots, F used in the text are defined in this figure. The inset shows a zoom of the region around T_{pl} . Note the small temperature fluctuations during the plateau period.

If the system had lost all specific memory of its stay at temperature T_{pl} , point F would be situated at point E translated downwards by the cumulative part of the memory, measured by $|C - D|$. The extra difference can thus be taken as a measure of the specific memory of the processes taking place at T_{pl} . We therefore define the memory indicator $\mathcal{M}'(\omega)$ as:

$$\mathcal{M}'(\omega) = |E - F| - |C - D|. \quad (4)$$

A situation where the memory is perfectly conserved would lead to $\mathcal{M}' = |A - B| \equiv \delta C'$.

As shown in Figure 4, this difference is positive in KTN, and again behaves as a power-law with frequency. Interestingly, the exponent describing this behaviour is very close to ν . For $T_{\text{pl}} = 12.1$ K and $t_w = 20\,000$ s, we find a value of 0.13, nearly identical to the value of ν reported in Figure 1. In the inset of Figure 4, we have shown the ‘memory ratio’, *i.e.* the ratio of \mathcal{M}' to the total isothermal decay $\delta C'$ during the plateau studied above, for different frequencies. This ratio is around 0.5 (showing that some memory is lost), but is nearly independent of frequencies, showing that the frequency dependence of the memory is actually that of the aging part of C' . A simple interpretation of this fact will be given below.

5 Interpretation

The microscopic origin of aging in disordered ferromagnets or ferroelectrics is most probably the slow dynamics of domain walls, pinned by impurities. The important ingredient to understand the physics of these pinned objects is the fact that the typical pinning energy associated with

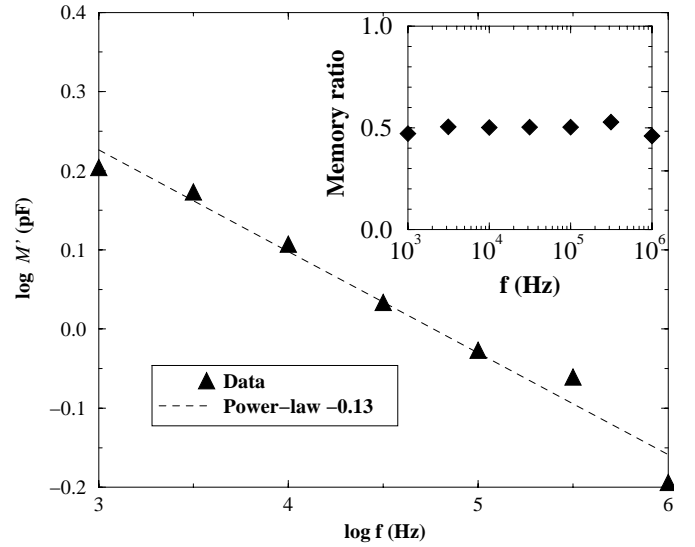


Fig. 4. The memory indicator $\mathcal{M}'(\omega)$ as a function of frequency for $T_{\text{pl}} = 12.1$ K and $t_w = 20\,000$ s, in a log-log plot. The exponent found here is the same as in Figure 1, as demonstrated in the inset where the ‘memory ratio’ $\mathcal{M}'/\delta C'$ is shown to be constant, around 0.5, over at least three decades in frequency.

a portion of wall of linear length ℓ grows as $\mathcal{Y}\ell^\theta$, where θ is a certain exponent and \mathcal{Y} an energy scale [28]. This means that the time needed to equilibrate such a portion of wall grows exponentially, as $\tau(\ell) \sim \exp(\mathcal{Y}\ell^\theta/T)$. Correspondingly, as emphasized in [16], time scales are strongly hierarchized, in the sense that $\tau(2\ell) \gg \tau(\ell)$. Hence, some small scale reconfigurations can take place while the overall, large scale configuration of the domain wall is fixed. This small scale motion between metastable states leads to better and better pinned configurations, and therefore a smaller susceptibility: this leads to aging, even if the overall size of the domains does not change. The large scale coarsening leads to a progressive increase of the average domain size R , and thus a decrease of the fraction of dipoles belonging to domain walls, as $1/R$. The coexistence of these two effects suggests that the *aging part* of the susceptibility (magnetic or dielectric) should read:

$$C_{\text{ag}}(\omega, t_w, T_{\text{pl}}) = \frac{1}{R(t_w)} [c_{\text{st}}(\omega, T_{\text{pl}}) + c_{\text{rec}}(\omega, t_w, T_{\text{pl}})], \quad (5)$$

where $c_{\text{st}}(\omega, T_{\text{pl}})$ is the equilibrium contribution of a wall of fixed (large) size, and $c_{\text{rec}}(\omega, t_w, T_{\text{pl}})$ is the aging contribution, due to small scale reconfigurations which allow the wall to find deeper and deeper metastable states. Simple ‘trap’ models for these reconfigurations suggest that $c_{\text{rec}} \propto (\omega t_w)^{-\nu}$ ($\nu < 1$) [29]. More general (subaging) forms are possible [16, 30], as observed experimentally (see Fig. 2). A possible physical mechanism is that each metastable state is visited a large number of times. Note that in Figure 2 we have neglected the time dependence of R , which is justified if the cooling rate $|\mathcal{R}|$ is small

enough. In that case, the relative growth of R during t_w can be neglected.

As explained in [16, 18, 29], the reformation contribution can account for the rejuvenation and memory effect. As the temperature is decreased, the small scale motion, which was in equilibrium at the higher temperature, is suddenly driven out of equilibrium. The aging part $c_{\text{rec}} \propto (\omega t)^{-\nu}$ has a singularity for $t = 0$ that reflects the excess dissipation that arises in strongly out of equilibrium situations. The physical mechanism is that some high energy traps are suddenly overpopulated at the lower temperature and must quickly empty out. Therefore any ‘micro-quench’ leads to an increased susceptibility and new aging dynamics. Simultaneously, larger scale dynamics is effectively frozen and contributes to memory. The distinctive feature of equation (5) is however the *cumulative* factor $1/R$ that always grows as soon as the system is in its ferroelectric phase (albeit more slowly at smaller temperatures). This cumulative factor is at the origin of all cooling rate effects (see the discussion in [21]). It also explains the change of sign of the slope \mathcal{S} reported above: at high frequency, the reformation contribution $c_{\text{rec}}(\omega, t_w, T_{\text{pl}})$ is negligible, and the main effect is the progressive increase of R which leads to a decrease of $C_{\text{ag}} \simeq c_{\text{st}}(\omega, T_{\text{pl}})/R(t)$. Therefore the slope \mathcal{S} just after the plateau is positive: the capacitance decreases when the temperature is lowered further. On the contrary, the rejuvenation effect is dominant at low enough frequencies, and explains why \mathcal{S} is negative. The frequency dependence of $\delta\mathcal{S}$ is thus expected to be similar to that of the isothermal decay of the capacitance, as was reported in Section 4.

It is important to note that the large scale domain motion is also responsible for the loss of memory, as suggested in [6]: if, during the period spent at lower temperatures, the domains have had enough time to move substantially and lose track of their previous position relative to the impurities, the memory effect is lost. More precisely, let us call $p(t_{\text{low}})$ the probability that a given wall has *not* substantially moved during the time $t_{\text{low}} = 2(T_{\text{pl}} - T_{\text{min}})/|\mathcal{R}|$ spent at low temperatures, and thus retains the memory of the past history. The capacitance reached at T_{pl} on the heating curve after a plateau at T_{pl} (point ‘F’ in Fig. 3) is therefore equal to:

$$C_{\text{pl}}(\omega) \simeq \frac{1}{R(t_w + t_{\text{low}})} [c_{\text{st}}(\omega, T_{\text{pl}}) + p c_{\text{rec}}(\omega, t_w, T_{\text{pl}}) + (1 - p) c_{\text{rec}}(\omega, 0^+, T_{\text{pl}})], \quad (6)$$

where the origin of time is chosen when T_{pl} is first reached (point ‘A’). The above equation means that a fraction p of the signal has kept full memory, while a fraction $1 - p$ is totally rejuvenated. This decomposition is only approximate, since the memory part is affected by the time spent near T_{pl} . The memory indicator defined above can be written as:

$$\mathcal{M}(\omega) = \frac{1}{R(t_w + t_{\text{low}})} [c_{\text{st}}(\omega, T_{\text{pl}}) + c_{\text{rec}}(\omega, 0^+, T_{\text{pl}})] - C_{\text{pl}}(\omega), \quad (7)$$

where the first term in the right hand side is the capacitance that one would measure in the absence of any memory ($p = 0$). We there find that the memory indicator \mathcal{M} is related to the isothermal decay of the capacitance $\delta C(\omega, t_w, T_{\text{pl}})$ by:

$$\mathcal{M}(\omega) \simeq \frac{p(t_{\text{low}})}{R(t_w + t_{\text{low}})} [c_{\text{rec}}(\omega, 0^+, T_{\text{pl}}) - c_{\text{rec}}(\omega, t_w, T_{\text{pl}})], \quad (8)$$

or

$$\mathcal{M}(\omega) = p(t_{\text{low}}) \frac{R(t_w)}{R(t_w + t_{\text{low}})} \delta C(\omega, t_w, T_{\text{pl}}). \quad (9)$$

This proportionality is indeed confirmed by Figure 4. Note that p is expected to decrease when t_{low} increases. On the other hand, the ratio $R(t_w)/R(t_w + t_{\text{low}})$ (which is expected to be close to 1 in the experiments) also decreases when t_{low} increases. Therefore, the amplitude of the memory is expected to decrease as the cooling rate decreases. We have checked this feature experimentally, and have found that the dependence of \mathcal{M}' on the cooling rate \mathcal{R} can be fitted approximately by a power law $\mathcal{M}' \propto \mathcal{R}^\beta$ with $\beta = 0.28$ for $T_{\text{pl}} = 14.4$ K and $t_w = 10\,000$ s, and \mathcal{R} in the range 1.3 to 20.8 mK/s. This experimental dependence suggests that for very small cooling rates, the memory effect is eventually totally erased by domain growth, as proposed in [6] for disordered ferromagnets.

6 Conclusion

The present experiments can be seen as a quantitative confirmation of the qualitative scenario of aging proposed in [6, 16] for disordered ferromagnets, which ascribes the observed effects to the motion of pinned domain walls. The simultaneous presence of overall domain growth and internal reformation modes allows one to account for the phenomenology of temperature cycling: cumulative effects and memory erosion are due to the former, whereas rejuvenation and memory are due to the latter, and therefore show up for small frequencies. We have also shown that all the quantities that measure the reformation contribution behave as power-laws of frequency, and that a subaging (ωt_w^μ) scaling holds, with a rather small value of the exponent $\mu \simeq 0.5$.

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